Study on the Effects of Palm Oil Fuel Ash (POFA) in Geopolymer Cement Properties

By

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the requirements for the
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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the

Petroleum Engineering Programme

Universiti Teknologi PETRONAS

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Approved by,

_____

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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 2013

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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SITI SHAHARA BINTI ZAKARIA

ABSTRACT

Geopolymerization is the current booming field of research for utilizing solid waste and by-products. It does give a cost-effective solution to many problems where hazardous residue has to be treated and stored at critical environmental conditions. From geopolymerization, a new technology which is geopolymer cement also known as green cement has been introduced.Geopolymer cement is also said to reduced greenhouse gas footprints when compared to conventional cement slurries used in oil and gas well cementing operations.

In this project, the writer has been assigned to carry out the study on geopolymer characteristic properties of Palm Oil Fuel Ash (POFA) and its effects as geopolymer cement materials. The main objective is to determine the relationship between compressive strength with curing time and other effecting factor such as alkaline solution concentration.Detailed study in the effects was carried out throught out the project.

The experiments will be done by mixing the POFA with other materials such as alkaline activators to produce cement paste. The cement paste is then poured into specific mould size and left for curing at different curing time. The results of compressive strength test are recorded and analysed.

In conclusion, the POFA geopolymer cement gives higher compressive strength in 12M of sodium hydroxide solution and with increment of alkaline activators to POFA mass ratio, the compressive strenght will increase.

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LIST OF ABBREVIATIONS

Al_2O_3	Aluminium Oxide
API	American Petroleum Institute
$CaCO_3$	Limestone
CaO	Calcium Oxide
CO_2	Carbon Dioxide
FA	Fly Ash
Fe_2O_3	Iron (III) Oxide
FTIR	Fourier Transform Infrared
K	Potassium
K_2O	Potassium Oxide
KOH	Potassium Hydroxide
LOI	Loss In Ignition
MgO	Magnesium Oxide
Na	Sodium
Na_2O	Sodium Oxide
Na_2SiO_3	Sodium Silicate
NaOH	Sodium Hydroxide
OPC	Ordinary Portland Cement
P_2O_5	Phosphorus Pentoxide
POFA	Palm Oil Fuel Ash
SiO_2	Silicon Dioxide

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Industrialization booming has been around late 18^{th} and 19^{th} centuries throughout the world. It is inevitable that industrialization brings a lot of positive impacts to the world, but it also leads to the generation and release of undesirable pollutants to the environment. The greatest problem faced by the industries is to dispose the waste products. Landfilling is not a desirable option because it creates huge financial problem to the foundries and also increases the threat to ground water contamination and environmental pollution. (Divya Khale, 2007)

Agricultural wastes however, are renewable and are found in abundance in many countries, as well in Malaysia. Utilization of these materials and the development of a domestic material industry based on the available materials should be encouraged. It is reported that the world palm oil production reached about 9.6 million tons per year in 1989 from a modest start in the early 1920s due to increasing demand for vegetable oil. Malaysia contributed 5.5 million tons per year from palm oil agriculture of 1.85 hectares during the same year, making it a major producer in palm oil. (Abdullah, 1997)

Currently, geopolymerization is a developing field of research for utilizing solid waste and by-products. It provides an advanced and cost effective solution to

industrial and agricultural problems which could be developed to manufacture precast structure and non-structural elements, concrete pavements and concrete products (Khale and Chaudry 2007). Therefore, it is one of the best methods to be used in handling the agricultural waste.

On the other hand, geopolymer usage in oil well cementing is a new technology which needs a proper study to determine the advantages of geopolymer cement compared to the conventional cement. This paper will be discussing on the study on the effect of Palm Oil Fuel Ash (POFA) in geopolymer cement properties to be used in oil well cementing. It is important to determine the compressive strength of the geopolymer cement using POFA with optimum curing temperature and time.

1.2 Problem Statement

Ordinary Portland Cement (OPC) is a main construction material used worldwide for oil well cementing. Unfortunately, manufacturing of OPC releases large amounts of CO_2 into the atmosphere and is responsible for 7-10% of total CO_2 emission in the world. (McLeod, 2005)

From an article published by Geopolymer Institute, OPC results from the calcination of limestone (calcium carbonate) at very high temperatures of approximately 1450-1500°C, and silico-aluminous material according to the reaction shows in the formula below:

$$5CaCO_3 + 2SiO_2 \longrightarrow (3CaO,SiO_2) + (2CaO,SiO_2) + 5CO_2$$

This shows that the manufacture of 1 metric tons of cement generates 1 metric tons of CO_2 greenhouse gas. (Geopolymer Institute)

According to technical paper produced by Ernest Orlando Lawrence Berkeley, National Laboratory, world cement demands and production are increasing significantly, leading to a growth in this industry's absolute energy use and CO_2

emission. Based on the report produced by World Business Council for Sustainable Development, the production of 1 metric ton (t) of cement releases estimated 0.73 to 0.99 of CO_2/t cement depending on the clinker-per-cement ratio and other factors. (Klee, 2009)

As noted above, more than 50 % of the CO2 released during cement manufacture, or approximately 540 kg CO2 per metric ton (t) of clinker, is from calcination in which limestone (CaCO3) is transformed into lime (CaO) in the following reaction, (Ali Hasanbeigi, 2012):

$$CaCO_3 \rightarrow CaO + CO_2$$

To reduce greenhouse gas emissions, efforts are needed to develop environmental friendly OPC. One such alternative is geopolymer cement. The geopolymer cement is estimated to reduce CO_2 emission depending on the types of materials used. (Davidovits, 2013)

However, there are limited researches done about geopolymer properties. According to the Geopolymer Institute, geopolymeric cements are manufactured in a different manner than Portland cement. Geopolymers do not depend on the calcination of calcium carbonate and thus do not release bounded CO_2. They also do not require extreme high temperature kilns, with large expenditure of fuel, nor do geopolymers require such a large capital investment in plants and equipment. The mechanical properties of these novel geopolymeric cements are similar to those of regular Portland cement. (Geopolymer Institute)

Therefore, further research about the geopolymer properties of cement based on POFA shall be carried out to determine the advantages of the POFA.

1.3 Objectives and Scope of Study

The objective of this paper is to study on the effect of POFA in geopolymer properties as cement material. The parameters that will be investigated are:

- Compressive strength
- Concentration and ratio of alkaline activators.

The scope of study is focusing on to determine and study the effects of the POFA as the raw materials for cementing and its cement properties. The results of geopolymer cement based on POFA will be compared to the conventional cement used in cementing activities in accordance to American Petroleum Institute specifications to ensure the compatibility of the cement with the oil and gas well conditions.

1.4 The Relevancy of the Project

Oil well cementing is important in oil and gas industry especially for the exploration and production process. The application of geopolymer cement for oil well cementing is still new, therefore a lot of studies need to be carried out to increase the findings of geopolymer technology compatibility with oil well conditions.

1.5 Feasibility of the Project within the Scope and Time Frame

This project is encompassing research and also laboratory work. Most of equipment and material are available in Drilling Simulation and Mud Preparation Laboratory at Petroleum Engineering Department. Whereas the geopolymer material used in this project, POFA is obtained from Kilang Sawit FELCRA Berhad Nasaruddin, Bota, Perak. This project can be done within 8 months given and the objectives can be achieved if the procedures are carefully followed.

CHAPTER 2

LITERATURE REVIEW

2.1 Geopolymer

Davidovits (1983) defined that the reaction of a solid aluminosilicate with a highly concentrated aqueous alkali hydroxide or silicate solution produces a synthetic alkali aluminosilicate material generally called a "geopolymer". Depending on the raw material selection and processing conditions, geopolymers can exhibit a wide range of properties and characteristics, including high compressive strength, low shrinkage, fast or slow setting, acid resistance, fire resistance and low thermal conductivity. Despite this wide range of commonly boasted attributes, these properties are not necessarily inherent to all geopolymeric formulations. (P. Duxson, 2006)

2.2 Geopolymerization

Geopolymerization is a geosynthesis (reaction that chemically integrates minerals) that involves naturally occurring silico-aluminates. Any pozzolanic compound or source of silica and alumina that is readily dissolved in the alkaline solution acts as a source of geopolymer precursor species and thus lends itself to geopolymerization.

According to Khale and Chaudary (2007), it is based on chemistry of alkali activated inorganic binders which discovered by a scientist named Purdon. Both

proposed that the hardening of the mechanism of alkali activated alumino silicate binder involves dissolution of Silicon (Si) or Aluminium (Al) in the presence of sodium hydroxide, and precipitation of calcium silicate or aluminium hydrate with the generation of sodium hydroxide. (Divya Khale, 2007)

Mechanism of geopolymers involves the polycondensation reaction of geopolymeric precursors i.e. alumino-silicate oxide with alkali polysiliates yielding polymeric Si-O-Al bond. Figure 1 below shows the conceptual model for geopolymerization. (P. Duxson, 2006)

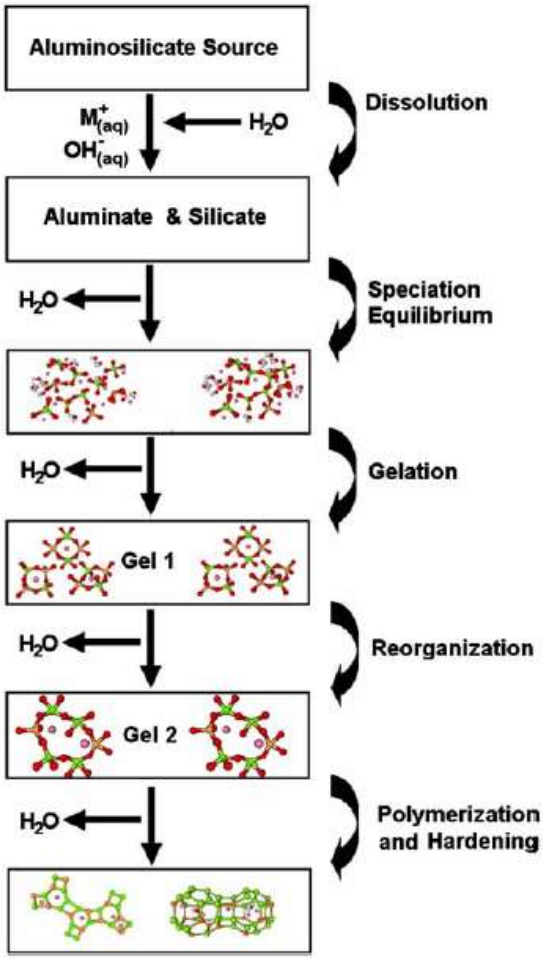


Figure 1: Conceptual model for geopolymerization

2.3 Geopolymer Cement

Geopolymer cement is a binding system that hardens at room temperature, like regular OPC. If a geopolymer compound requires heat setting it may not be called geopolymer cement but rather geopolymer binder. (Davidovits, 1991)

Geopolymer cement is an innovative material and a real alternative to conventional OPC for use in constructions and offshore applications. It relies on minimally processed natural materials or industrial by-products to significantly reduce its carbon footprint, while also being very resistant to many of the durability issues that can surpass conventional concretes.

Creating geopolymer cement requires an alumina silicate material, a user-friendly alkaline reagent (sodium or potassium soluble silicates with a molar ratio MR SiO_2:M_2O>1.65, M being Na or K) and water. Room temperature hardening relies on the addition of calcium cations, essentially iron blast furnace slag.

Geopolymer cements cure more rapidly than OPC. They gain most of their strength within 24 hours. However, they set slowly enough that they can be mixed at a batch plant and delivered in a concrete mixer. Geopolymer cement also has the ability to form a strong chemical bond with all kind of rock-based aggregates. (Davidovits, 2013)

On March 2010, the US Department of Transportation Federal Highway Administration released a TechBrief titled Geopolymer Concrete states that: The production of multipurpose, cost-effective geopolymer cements that can be mixed and hardened essentially like OPC represents a game changing advancement, developing the construction of transportation infrastructure and the building industry. (Dam, 2010)

There is often confusion between the meanings of the two terms 'geopolymer cement' and 'geopolymer concrete'. A cement is a binder whereas concrete is the composite material resulting from the addition of cement to stone aggregates. In

other words, to produce concrete one purchase cements (generally OPC or geopolymer cement) and adds it to the concrete batch. (Davidovits, 2013)

Figure 2 below shows the components of geopolymer cements.

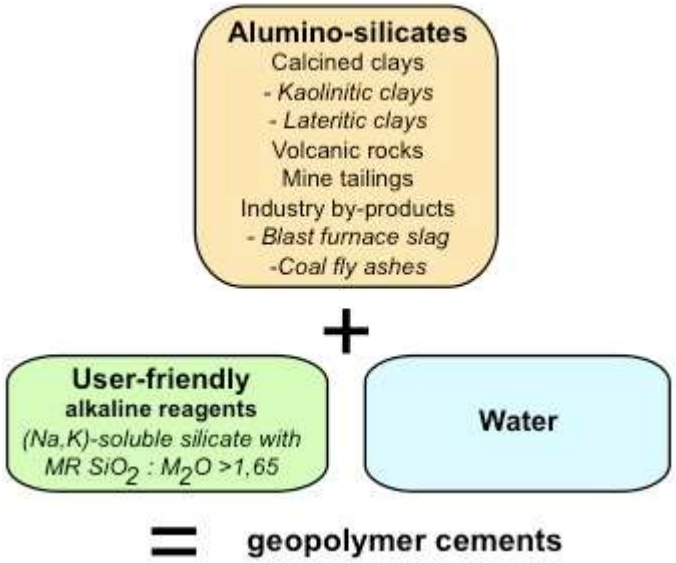


Figure 2: Geopolymer cement compositions (*Source:* (Davidovits, 2013))

2.4 Ordinary Portland cement (OPC)

OPC is the most commonly used type of cement in the world today. Portland cement is a hydraulic material, which requires the addition of water in order to form exothermic bonds, and is not soluble in water. Initially designed as a cement which would set slowly, allowing enough time for it to be properly laid, and a water resistant cement which could be used in construction applications where water would come in contact with the cement. OPC was first patented in 1824 by an English man, Joseph Aspdin, but the mix which became truly successful, and which is still used nowadays, was designed by his son, William Aspdin in around 1843. (Raw Polymers Ltd)

2.5 Cement properties

2.5.1 Compressive Strength

According to Instron glossary, compressive strength is maximum stress a material can sustain under crush loading. The compressive strength of a material that fails by shattering fracture can be defined within fairly narrow limits as an independent property. Nevertheless, the compressive strength of materials that do not shatter in compression must be defined as the amount of stress required to distort the material an arbitrary amount. Compressive strength is calculated by dividing the maximum load by the original cross-sectional area of a specimen in a compression test. For an oil well cement, the cement sheath must developed the minimum compressive strength at least 500 psi, which is almost equal to 3.45 Mpa. (Faizal M.N, 2007)

2.5.2 Curing Temperature and Time

Curing is the maintenance of a satisfactory moisture content and temperature in concrete for a period of time immediately following placing and finishing so that the desired properties may develop. The need for adequate curing of concrete cannot be overemphasized. Curing has a strong impact on the properties of hardened concrete; proper curing will increase durability, strength, water tightness, abrasion resistance, volume stability, and resistance to freezing and thawing and deicers. Exposed slab surfaces are mainly sensitive to curing as strength development and freeze-thaw resistance of the top surface of a slab can be reduced significantly when curing is defective. (Cement Concrete & Aggregated Australia)

With proper curing, concrete becomes stronger, more impermeable, and more resistant to stress, abrasion, and freezing and thawing. The improvement is rapid at early ages but continues more slowly thereafter for an indefinite period.

Figure 3, 4 and 5 below shows the strength gain of concrete with age for different moist curing periods and the relative strength gain of concrete cured at different temperatures respectively. (Kosmatka S.H et al., 1988)

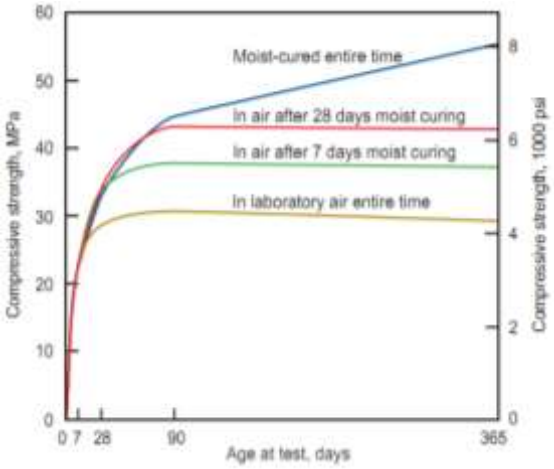


Figure 3 Effect of moist curing time on strength gain of concrete *(Source: Gonnerman & Shuman 1928,* (Kosmatka S.H et al., 1988)*)*

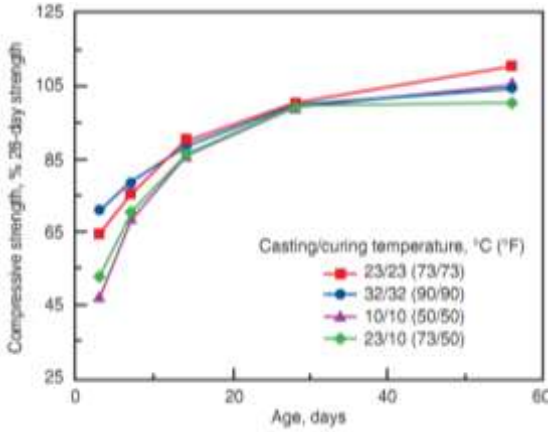


Figure 4 Effect on curing temp. on strength gain relative to 28-day strength *(Source: Gonnerman & Shuman 1928,* (Kosmatka S.H et al., 1988)

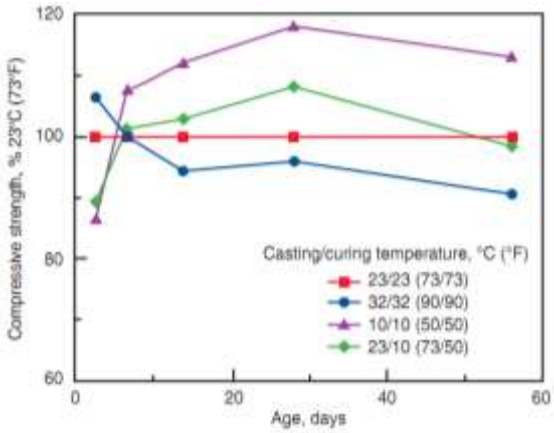


Figure 5: Effect of curing temperature on strength gain relative to strength of concrete at 23 degree Celcius ***(Source: Gonnerman & Shuman 1928,*** **(Kosmatka S.H et al., 1988)**

2.5.3 pH

pH is the most significant factor controlling the compressive strength of cements. The setting time of cement decreased as the pH of the activating solution increased. At lower pH values of the geopolymeric mix remained viscous and behave like cement while at higher pH, the mix achieved a more fluid gel composition, which is less viscous and is more workable. Strength at pH 14 was 50 times larger than those at pH 12 (less than 10 Mpa at pH 12, 50 Mpa at pH 14) of geopolymeric matrix utilizing cement as setting additive. Higher solubility of monomers was expected by KOH than NaOH because of higher alkalinity. With increasing pH, there was a predominance of smaller chain oligomers and monomeric silicate available to react with soluble aluminum. Further with increase in pH soluble aluminum increases and reacts with calcium available for reaction. Lower pH-value of the solution leads to lower monomer concentration.

The pH level of the activating solution strongly influences the final cement performance. Khale and Chaudhary reported that the strength measured from samples of pH 14 was five times greater than samples formed from pastes of pH 12; and they concluded that a pH range of 13–14 was most suitable for the formation of geopolymers with higher mechanical strength. (Divya Khale, 2007)

2.5.4 Alkali Concentration

Alkali concentration is the most significant factor for geopolymerization. The solubility of aluminosilicate increased with increasing hydroxide ion concentration. Higher concentration of NaOH yielded high compressive strength. 10 N KOH showed the highest strength of 60 Mpa, but the strength decreased on increasing the KOH concentration from 10 N to 15 N, probably due to excess K^+ ions in the framework. Addition of K_2O was found to benefit the compressive strength and also to reduce the occurrence of cracking. High NaOH addition accelerated chemical dissolution but depressed ettringite and CH formation. An excess of OH^- concentration in the system decreased the strength of the system. Higher the alkalinity of the hydration water, slower the rate of the hydration during the binder hydration. (Divya Khale, 2007)

The most common alkaline activators are hydroxides of sodium and potassium (NaOH and KOH). The highest observed mechanical strengths have resulted from the use KOH in varying concentrations. Since K^+ is more basic, it allows a higher rate of solubilized polymeric ionization and dissolution leading to a dense polycondensation reaction that provides greater overall network formation and an increase in the compressive strength of the matrix. Conversely, a study by Arjunan et al. discovered that sodium hydroxide in low concentration was the most effective chemical activator for low calcium fly ash. (Petermann et al., 2010)

2.6 Classification of Cement

The cement type are characterize according to the API classification as published in API Standards 10 entitled, "Specification for Oil-Well Cement and Cement Additives."

Table 1 below shows the API classification of cement.

Table 1: API classification of cement (Source: (Smith, 1987))

API Classification	Mixing Water (gal/sack)	Slurry Weight (lbm/gal)	Well Depth (ft.)	Static Temperature (^{o}F)
A (Portland)	5.2	15.6	0 to 6,000	80 to 170
B (Portland)	5.2	15.6	0 to 6,000	80 to 170
C (high early)	6.3	14.8	0 to 6,000	80 to 170
D (retarded)	4.3	16.4	6,000 to 12,000	170 to 260
E (retarded)	4.3	16.4	6,000 to 14,000	170 to 290
F (retarded)	4.3	16.2	10,000 to 16,000	230 to 320
G (basic)**	5.0	15.8	0 to 8,000	80 to 200
H (basic)**	4.3	16.4	0 to 8,000	80 to 200

** Can be accelerated or retarded for most well conditions

Besides the specific API classification cements, there are a number of cementitious materials used very effectively for cementing wells. These materials include (1) pozzolanic-Portland cements, (2) pozzolan-lime cements, (3) resin or plastic cements, (4) gypsum cements, (5) diesel oil cements, (6) expanding cements, (7) refractory cements, (8) latex cement and (9) cement for permafrost environment. (Smith, 1987)

Other than that, there are a few physical properties of API cements, as stated in the Table 2 below:

Table 2: Physical properties of API cements (PetroWiki, 2012)

				A	B	C	G	H
Well cement class:								
Mix water, wt% of well cement:				46	46	56	44	38
Fineness tests (alternative methods):								
Turbidimeter (specified surface, minimum, m_2/kg):				150	160	220	—	—
Air permeability (specified surface, minimum, m_2/kg):				280	280	400	—	—
Free-fluid content, maximum, mL:				—	—	—	3.5	3.5
Compressive-strength test, 8-hour curing time	Schedule number, Table 7	Curing temp., °F (°C)	Curing pressure, psi (kPa)	Minimum Compressive Strength, psi (MPa)				
	—	100 (38)	Atmos.	250 (1.7)	200 (1.4)	300 (2.1)	300 (2.1)	300 (2.1)
	—	140 (60)	Atmos.	—	—	—	1,500 (10.3)	1,500 (10.3)
Compressive-strength test, 24-hour curing time	Schedule number, Table 7	Final curing temp., °F (°C)	Final curing pressure, psi (kPa)	Minimum Compressive Strength, psi (MPa)				
	—	100 (38)	Atmos.	1,800 (12.4)	1,500 (10.3)	2,000 (18.8)	—	—
Pressure/ temperature thickening-time test	Specification test schedule number, Table 10		Maximum consistency, 15 to 30 min stirring period, B_c	Minimum Thickening Time, min				
	4		30	90	90	90	—	—
	5		30	—	—	—	90	90
	5		30	—	—	—	120 max.	120 max.

B_c = Bearden units of consistency, obtained on a pressurized consistometer, as defined in Sec. 9 of API *Spec. 10A* and calibrated as per the same section.[8]

2.7 Overview of Palm Oil Industry in Malaysia

Over the last few decades, the Malaysia palm oil industry has grown to become a very important agriculture-based industry. During the late 1950s the expansion of the industry started as part of government's diversified cautious policy from rubber to oil palm and also to raise the socio-economic status of the expanding population in the country.

Today, Malaysia has become the world's largest producer and exporter of palm oil, replacing Nigeria as the chief producer since 1971 as shown in Figure 5 below. Malaysia is blessed with favorable weather conditions which prevail throughout the year which is advantageous for palm oil plantation. Thus, it is not surprising that the highest yields have been obtained from palms grown in this region, which is far from its natural habitat. (Abdullah, 1997)

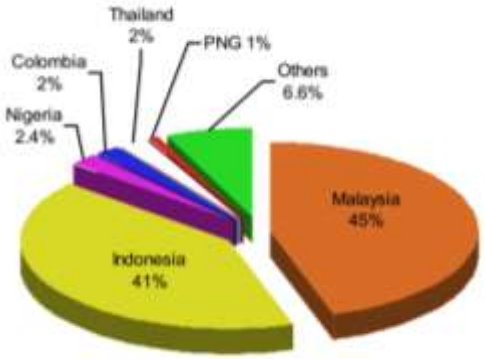


Figure 6: List of world main palm oil producer (Source: (Yacob, 2007))

As for the palm oil tree, the average economic life-span is 25 years. Therefore, POFA are available daily throughout the year when the palm are pruned during the harvesting of fresh fruit bunches for the production of oil. (Hassan O.A et al., 1996)

Table 3 below shows the availability of oil-palm trunks and frond (mt, dry matter basis) in Malaysia.

Table 3: Estimated availability of oil-palm trunks and frond (mt, dry matter basis) in Malaysia (*Source: Mohamad H, 1986*)

Year	Trunks	Fronds		Total
		Replanting	Pruning	
1990	1.32	0.25	16.92	18.49
1992	2.39	0.64	17.64	21.67
1994	4.60	0.88	17.89	22.37
1996	4.36	0.83	19.09	24.28
1998	7.48	1.42	18.18	27.08
2000	7.02	1.34	17.85	26.21

As stated in Beijing Forestry and Parks Departments of International Corporation, Malaysia is amongst the world's top producers of palm oil with the current planted area is expanding to around 4.0 million hectares. Success of palm oil industry in Malaysia is from the confluence of government and private sector strategies and policies. In spite of the huge production, the oil consists of only about 10% of the total biomass produced in the plantation. The balance consists of huge amount of oil palm wastes such as oil palm shells, mesocarp fibers and empty fruit bunch (from the mills) and oil palm fronds and oil palm trunk (from the field during replanting).

2.8 Palm Oil Fuel Ash (POFA)

Palm Oil Fuel Ash also known as POFA is a by-product of palm oil factory. After palm oil is extracted from the palm oil fruit, both palm oil husk and palm oil shell are burned to be used as fuel in the boiler of palm oil factory. Generally, after combustion about 5% palm oil fuel ash by weight of solid wastes is produced. (Vanchai Sata, 2004)

The physical characteristic of POFA is very much influenced by the operating system in palm oil factory. According to Sumadi and Hussin (1995), POFA produced in Malaysian palm oil mill is discarded as waste without any profitable return. POFA is considered as a nuisance to the environment. Since Malaysia is continuous to increase production of palm oil, therefore more ashes will be produced. (K.Abdullah, 2006)

Therefore, the usage of POFA as the geopolymer material in this project may solve the environmental problems as well as to find another function of it.

Table 4 below depicts the chemical composition of POFA.

Table 4: Chemical composition (%) of POFA (M. Azreen, 2011)

Type	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**Na_2O**	**K_2O**	**P_2O_5**	**LOI**
POFA	53.5	1.9	1.1	8.3	4.1	1.3	6.5	2.4	18.0

Where;

SiO_2	Silicon Dioxide
Al_2O_3	Aluminium Oxide
Fe_2O_3	Iron (III) Oxide
CaO	Calcium Oxide
MgO	Magnesium Oxide
Na_2O	Sodium Oxide
K_2O	Potassium Oxide
P_2O_5	Phosphorus pentoxide
LOI	Loss in Ignition

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

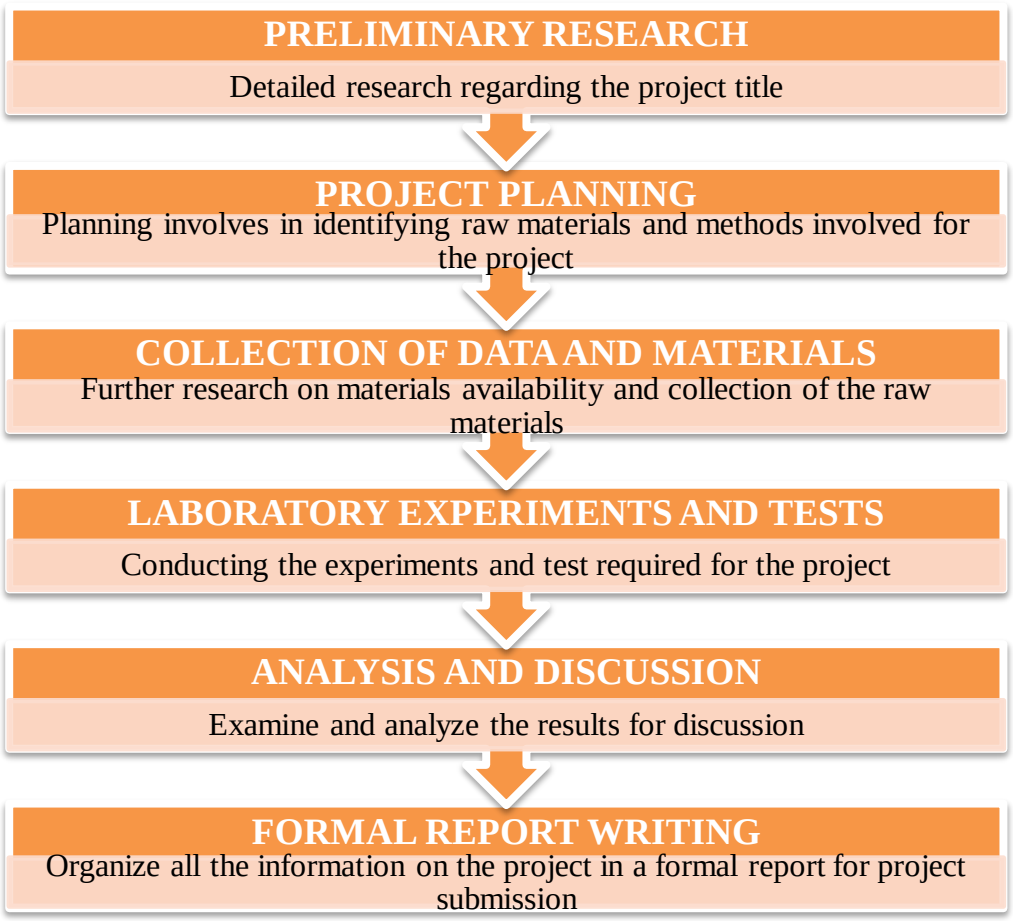
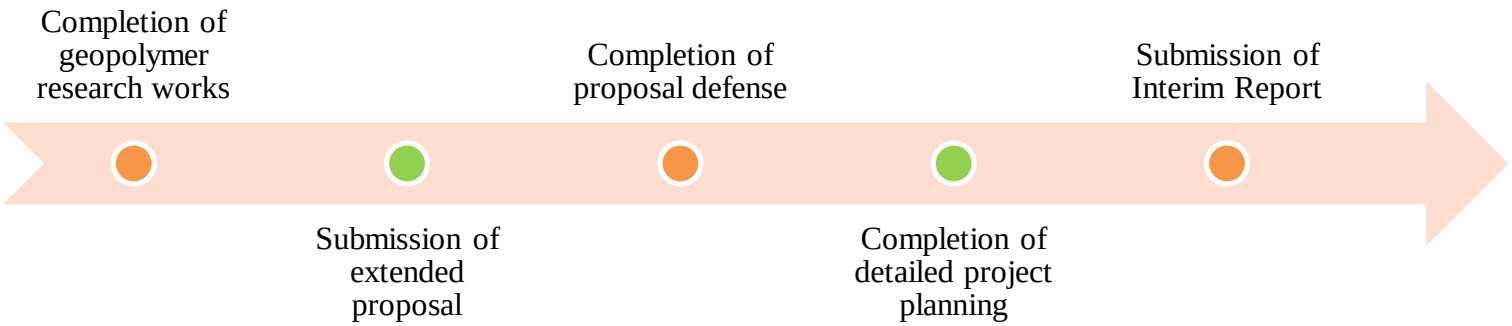


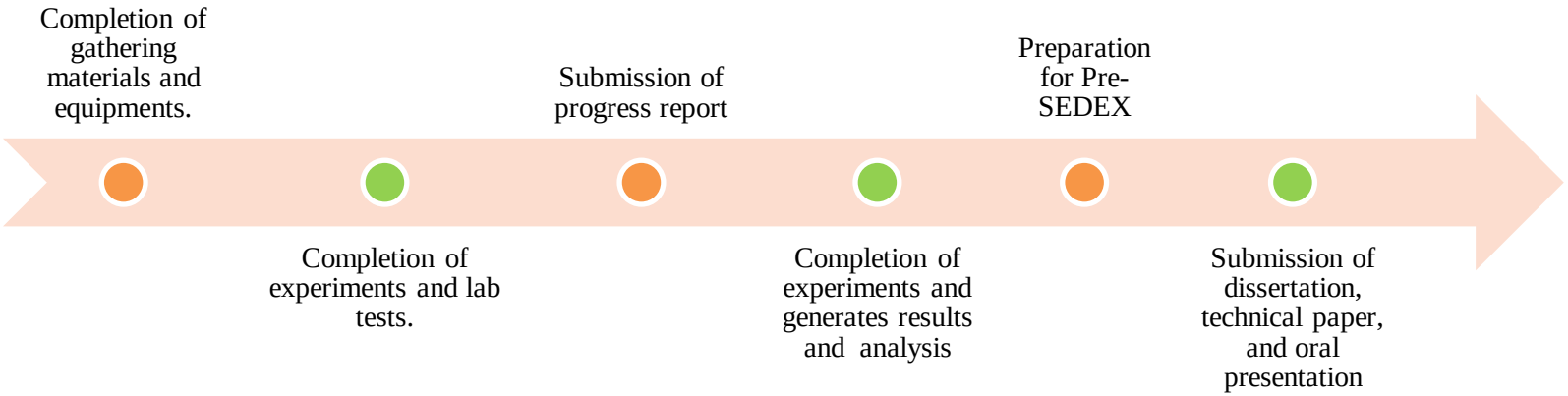
Figure 7: Research methodology chart

3.2 Key Milestone and Project Activities

FYP I



FYP II



3.3 Gantt Chart

PROJECT ACTIVITIES	WEEKS																												
	Final Year Project 1														Final Year Project 2														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Topic Selection and Proposal																													
Preliminary Research Work																													
Submission of Extended Proposal																													
Identify material and equipment																													
Proposal Defense																													
Project Work Detailed Planning																													
Submission of Interim Draft Report																													
Submission of Interim Report																													
Gathering Materials																													
Conducting Experiments and Lab Tests																													
Submission of Progress Report																													
Result Analysis and Discussion																													
Pre-SEDEX Preparation																													
Submission of Draft Report																													
Submission of Dissertation *(soft bound)*																													
Submission of Technical paper																													
Oral presentation																													
Submission of Project Dissertation *(hard bound)*																													

The above Gantt chart shows the proposed activities for the project during FYP I and FYP II. Based on the Gantt chart, the project is feasible in the given time frame. The blue box is showing the project works whereas the red box is showing the datelines of submission for the project works.

3.4 Material and Procedure

Table 5: List of materials and equipment used

Materials	Equipment
Palm Oil Fuel Ash (POFA) Fly Ash (FA) Sodium Silicate solution (Na_2SiO_3) Sodium Hydroxide solution (NaOH) (10M, 12M, 15M)	Cement moulds Grinding machine Weighing machine Constant Speed Mixer Roller Oven Compressive Strength Tester

3.4.1 POFA Geopolymer Cement Procedures:

1. Prepare a suitable amount of POFA by drying and sieving into finer particles.
2. Weight the POFA to desired weight.
3. Prepare 1 part of 10M of sodium hydroxide with POFA weight as reference.
4. Prepare 2.5 parts of sodium silicate solution to the mass of the sodium hydroxide solution.
5. Mix the sodium silicate solution together with sodium hydroxide solution in the mixer cup.
6. Mix the POFA with alkaline solution for 10 minutes at 4000 rpm.
7. Add in 10 % of water (based on ash powder weight) to give workability of the cement slurry.
8. Fill up the cement slurry into 50 mm X 50 mm cement mould.
9. Mould should be tightly covered with aluminium foil. Cure the cement at 60˚C for 24 hours.
10. Cure the batches of cement at ambient temperature for 3 day, 7 days and 14 days.
11. Repeat the experiment with 12M and 15M of sodium hydroxide solution.
12. Table 6 shows the mix proportions used during the experiment.

Table 6 Mix proportions of POFA geopolymer cement

Mix Composition	Mix proportion (kg/m^3)				Mass Ratio	
	POFA	FA	Na_2SiO_3	NaOH	Alkaline activators	Ash Powder
100 % POFA	1576	-	451	180	0.4	1.0
100 % POFA	1576	-	676	270	0.6	1.0
100 % POFA	1576	-	901	360	0.8	1.0
25 % POFA : 75 % FA	392	1184	451	180	0.4	1.0

3.4.2 Compressive Strength Testing Procedures:

1. Measure the dimensions of the surface in which the load is to be applied. Let it be 'L' and 'W' respectively. Since it is standardized mold of 50 mm X 50 mm, the cross-section area is constant at 2500 mm^2.
2. Place the cube in compressive testing machine and apply load uniformly at the rate of 4000N/mm^2.
3. Note the load at which the cube fails. Let it be 'P'.
4. Calculate the compressive strength of the cube.
5. Repeat the same procedure with the remaining 2 cubes.
6. 3 specimens should be tested and its average should be taken as its final compressive strength.
7. The calculation of compressive strength is done by using the following formula.

$$\sigma_i = \frac{F_i}{A_i}$$

Where;

σ_i = the compressive strength, N/mm^2

F_i = the maximum load, N

A_i = the cross-section area at which load is applied, mm^2

Figure 8: Compressive Strength Tester

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Results

This chapter will discuss the results obtained from the experiment done. The test results were presented into tables and figures which will further elaborated in this chapter.

4.1.1 Compressive Strength Test Result:

Table 7 Compressive Strength Test result for 100 percent POFA geopolymer cement

NaOH Concentration	Compressive strength (Mpa)		
	3d	7d	14d
10 M	0.62 0.65 0.63	0.74 0.52 0.75	0.91 0.75 0.52
Average	0.63	0.67	0.73
12 M	0.65 0.72 0.73	0.84 0.94 0.89	0.93 0.95 0.88
Average	0.70	0.89	0.92
15 M	0.52 0.53 0.49	0.86 0.89 0.73	0.89 0.88 0.94
Average	0.51	0.83	0.90

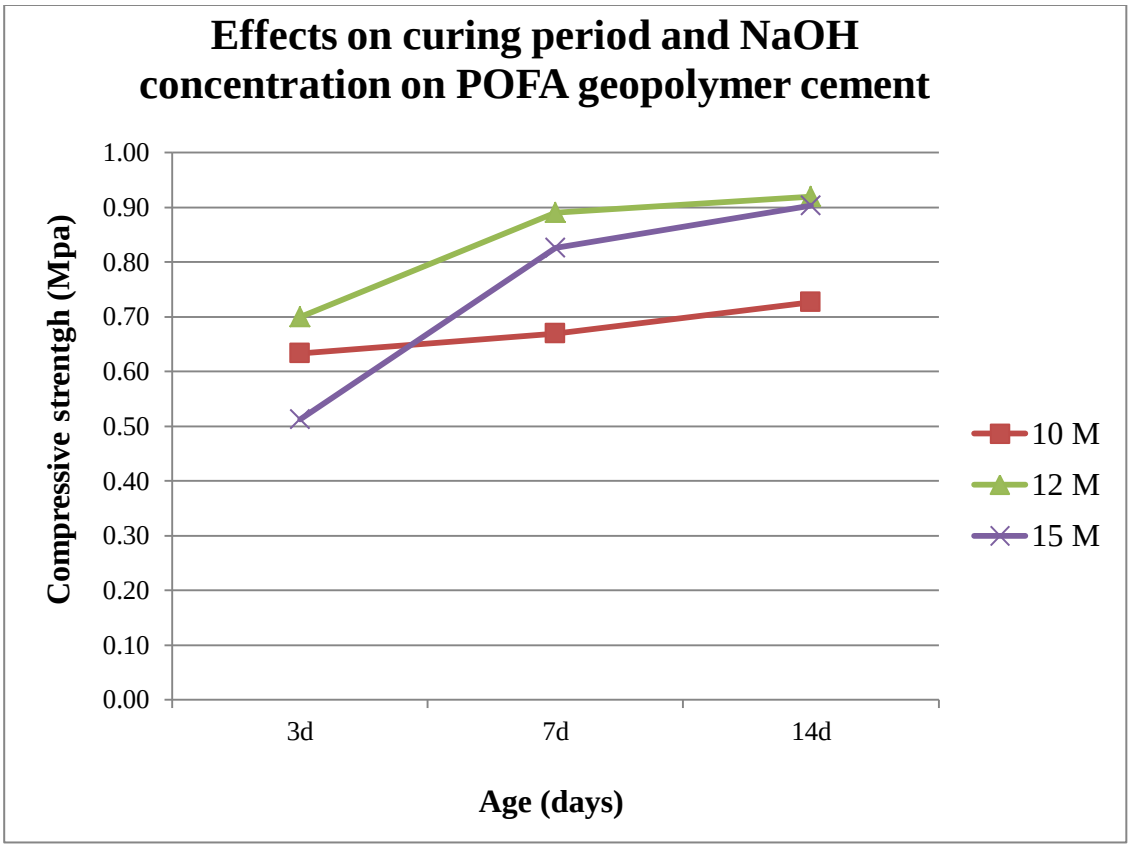


Figure 9 Graph on the effects on curing time and NaOH concentration on POFA geopolymer cement

Figure 8 above shows the effects between curing time and sodium hydroxide concentration on POFA geopolymer cement compressive strength.

For 3 day of curing and 10M of sodium hydroxide solution showed the compressive strength of 0.63 Mpa. Whereas, for the 7 days of curing time, the compressive strength obtained is 0.67 Mpa. The compressive strength result increases to 0.73 Mpa for 14 days of curing time.

For 12M of sodium hydroxide solution concentration, the compressive strength obtained for 3 day curing time is 0.70 Mpa. The compressive strength obtained for 7 days curing time is 0.89 Mpa while the compressive strength recorded for 14 days curing time is 0.92 Mpa.

The compressive strength results for 15M of sodium hydroxide solution concentration, for 3 days, 7 days and 14 days curing time are 0.51 Mpa and 0.83 Mpa and 0.90 Mpa respectively.

From Figure 8, it shows that the compressive strength is increasing from 3 days 14 days curing time for all sodium hydroxide solution concentrations. The compressive strength also increased from 10M to 12M of sodium hydroxide solution. However, the compressive strength decreases when using 15M of sodium hydroxide solution.

The experiment is repeated using different mix composition of geopolymer cement. The curing time is kept constant at 3 days, whereas the sodium hydroxide concentration is varies using 10M, 12M and 15M. The results are presented in the Table 7 and Figure 9 below.

Table 8 Compressive strength test for different composition of geopolymer cement

NaOH Concentration	**Compressive strength (Mpa)**	
	Geo Cement A 100 : 0 (POFA :FA)	**Geo Cement B 25 : 75 (POFA : FA)**
10 M	0.62 0.65 0.63	1.04 0.99 1.02
Average	0.63	1.02
12 M	0.65 0.72 0.73	1.20 1.19 1.39
Average	0.70	1.26
15 M	0.52 0.53 0.49	1.20 1.18 1.24
Average	0.51	1.21

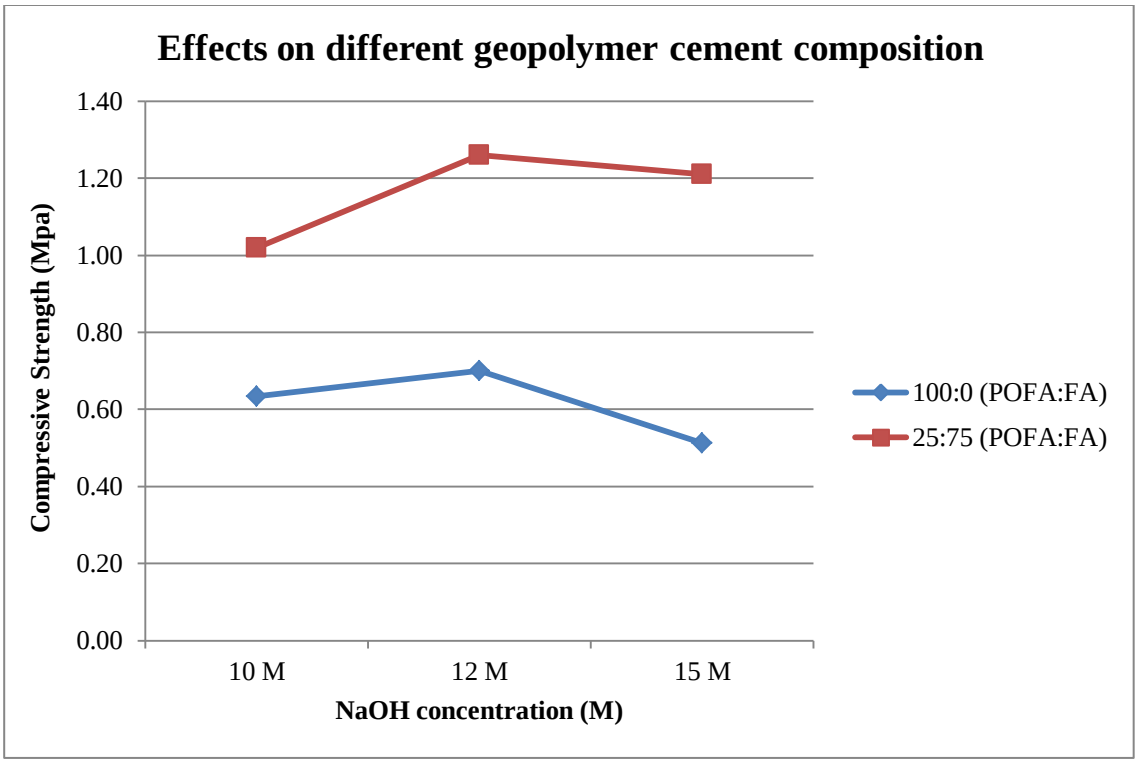


Figure 10 Effects on different geopolymer cement composition on POFA geopolymer cement

Figure 9 depicts the effects on different geopolymer cement composition and sodium hydroxide on its compressive strength. The new cement composition is using 25:75 ratio of POFA and Fly ash.

The compressive strength of Geo Cement A which is using 100% POFA and10M, 12M, 15M of sodium hydroxide is recorded at 0.63 Mpa, 0.70 Mpa and 0.51 Mpa respectively.

The Geo Cement B which using 25% of POFA added with 75% of Fly Ash has higher compressive strength of 1.02 Mpa when using 10M of sodium hydroxide solution. The compressive strength increases to 1.26 Mpa when increasing the molarity concentration of sodium hydroxide to 12M. The compressive strength recorded for Geo Cement B using 15M of sodium hydroxide solution is 1.21 Mpa.

From the graph shown is Figure 9, the compressive strength is increases when fly ash is added into the cement mix composition. The compressive strength also increases as the concentration of sodium hydroxide solution is increases from 10M to 12M for both geopolymer cement compositions. It shows that the addition of fly ash helps in improving the cement properties of POFA geopolymer cement.

The third test is done by manipulating the alkaline solution to POFA ratio. The ratio of alkaline solution to POFA ratio used is 0.4: 1.0, 0.6:1.0 and 0.8:1.0.

Table 9 Compressive strength test result for different alkaline solution to ashes ratio

Alkaline solution ratio	**Compressive strength (Mpa)**
0.4	0.65 0.72 0.73
Average	0.70
0.6	0.83 0.80 0.68
Average	0.77
0.8	0.84 0.79 0.86
Average	0.83

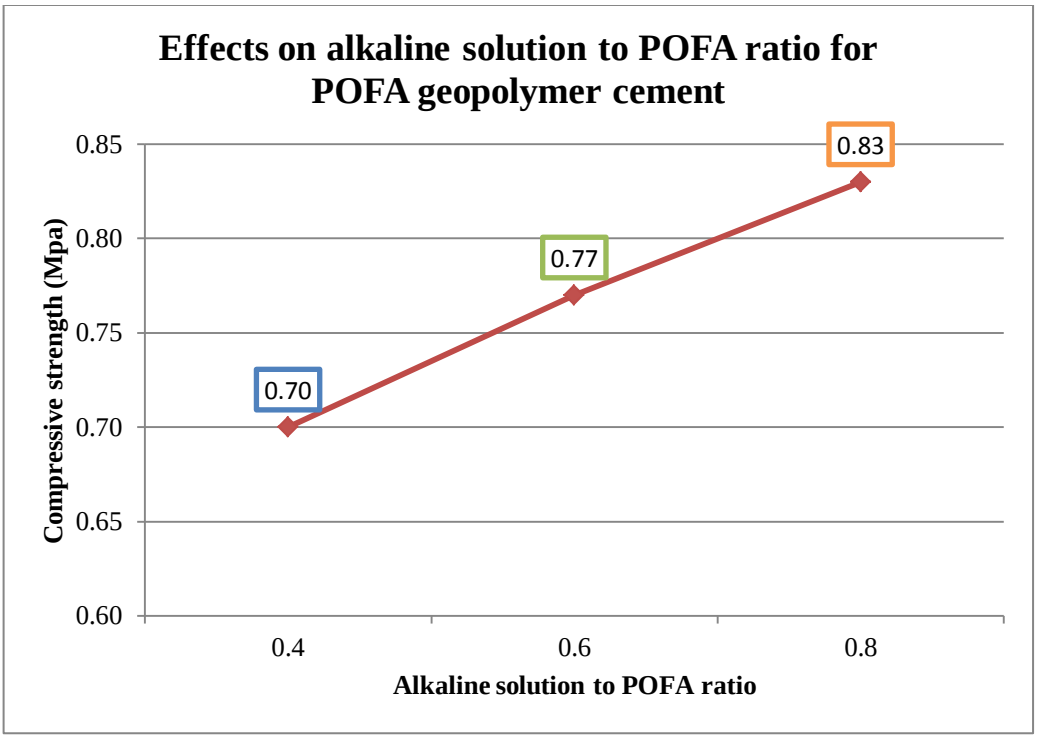


Figure 11 Effects on different alkaline ratio on the compressive strength of POFA geopolymer cement

The experiment is done within 3 days curing time. The sodium hydroxide concentration used in this experiment is kept constant at 12M.

The compressive strength test result recorded at 0.4:1.0 ratio is 0.70 Mpa. The compressive strength increases to 0.77 Mpa and 0.83 Mpa when the alkaline solution to POFA ratio increases to 0.6:1.0 and 0.8:1.0 correspondingly.

Figure 10 shows that the compressive strength is increasing proportionally as the alkaline solution to POFA ratio increases.

4.1.2 Fourier Transform Infrared (FTIR) Test Result

Fourier Transform Infrared (FTIR) test is done to identify unknown materials, quality or consistency of a sample and can determine the amount of components in a mixture.

For this project, POFA and FA samples were sent to the lab for the FTIR test. Figure 12 and Figure 13 below show the result of FTIR test for both POFA and FA.

From the FTIR test and IR Spectroscopy table (attached in the Appendix section), the compound present in both raw materials are summarized in the Table 10 below.

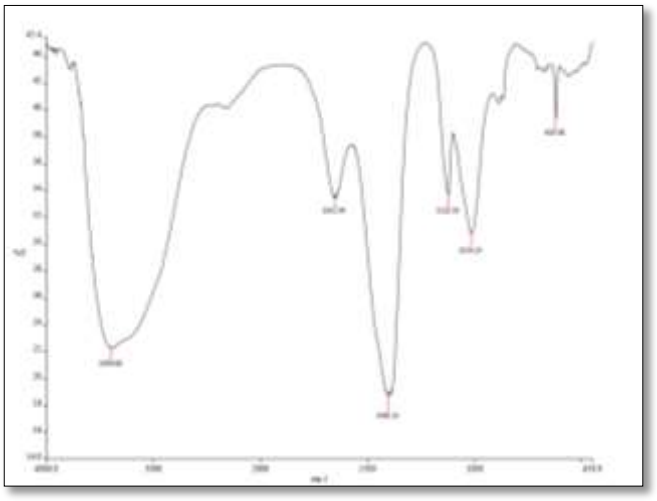


Figure 12 FTIR test result of POFA raw samples

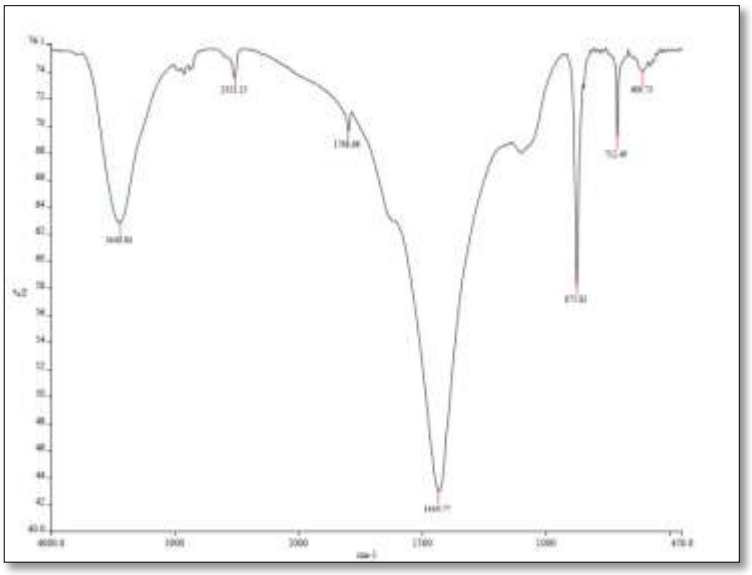


Figure 13 FTIR test result of FA raw samples

Table 10 Summary of FTIR test result

Types	Wave number (cm^{-1})	Functional group	Molecular motion
POFA	3390.08	Alcohols	O-H stretch
	1652.59	Alkenes	C=C stretch
	1403.23	Alkenes	C-H in-plane bed
	1122.50	Alcohols	C-O stretch
	1014.24	Alcohols	C-O stretch
	620.08	Alkynes	Acetylenic C-H bend
FA	3440.84	Alcohols	O-H stretch
	2513.25	Carboxylic acid	O-H stretch
	1789.69	Acid chlorides	C=O stretch
	1434.77	Aromatic	C-C stretch
	875.83	Alkenes	=C-H bend
	712.49	Aromatics	C-H oop
	609.73	Alkynes	Acetylenic C-H bend

4.2 Discussion

4.2.1 Various Molarity of Sodium Hydroxide Solution

The ultimate objective of this project is to determine the compressive strength of POFA geopolymer cement and compare the results with the API specifications for oil well cement. The method used in identifying the compressive strength has been determine by producing the POFA geopolymer cement cubes and test its compressive strength.

From the results tabulated in Table 6, the compressive strength increases when increasing the molarity of sodium hydroxide solution from 10M to 12M. However, after the 12M of sodium hydroxide solution, decreases in compressive strength can be observed. This result is in agreement with the results of Alonso and Palomo (2011) study, which also found that a 12M sodium hydroxide solution produced better results than the higher sodium hydroxide solution concentration. (Al Bakri et al., 2011)

On the other hand, research done by Hardjito et al. (2008) observed that, the compressive strength is increasing proportionally with increase in concentration of sodium hydroxide. This might due to different source materials used. Hardjito used low calcium fly ash (ASTM Class F) whereas Alonso and Palomo (2001) used a high purity metakaolin. The result may vary due to these differences. (Hardjito et al., 2008)

4.2.2 Different Geopolymer Cement Composition

The compressive strength obtained from this experiment shows very low strength of the POFA geopolymer cement. This is however expected might due to different materials composition used from other study.

Nevertheless, by addition of Fly Ash into the geopolymer cement composition, the compressive strength is improved. This is shown in the third test of this experiment whereas the compressive strength of Geo Cement B using 25:75 ratio of POFA/Fly ash is increased from 0.70 Mpa to 1.26 Mpa at 12M sodium hydroxide solution concentration and 3 days curing time. The result obtained is corresponding with study done M. Azreen et al., (2011) which found out that the compressive strength of POFA geopolymer cement with addition of FA is improved at the blended ash ratio of 70:30 ratio. (M. Azreen et al., 2011)

Aside from that, when referring to the FTIR test results, we can observed that the FA has additional functional group such as Carboxylic acid and Aromatics. An aromatic compound define as the arrangement of carbon atoms in a ring which gives the compound higher bond strength compared to linear arrangement of same number of carbons, therefore we may conclude that the presence of aromatic in FA does gives impact to the geopolymer cement by improving the compressive strength.

In conclusion, the geopolymer cement using 100% POFA show very low compressive strength which may be attributed to very slow chemical reaction due to less alumina in POFA. The alumina content is POFA is 1.9 %, whereas in alumina content in FA is 35.9 %.

4.2.3 Different Mass Ratio of Alkaline Solution to Ash Powder

The results of compressive strength with different alkaline activator/POFA mass ratio are shown in Table 8 and Figure 9. From the results, it shows the compressive strength is increasing with the increment of the ratio. The compressive strength of POFA geopolymer cement increases from 0.70 Mpa to 0.83 Mpa when increasing the alkaline activator/POFA ratio from 0.4 to 0.8.

Hardjito et al., (2004) concluded that compressive strength increases as a result of increase in sodium oxide content, which is mainly required for the geopolymerization reaction. (Hardjito et al., 2004)

4.2.4 Curing Time

In this study, it shows that the compressive strength of POFA geopolymer cement is gradually increases with extended curing time. The increase in compressive strength can be observed at all tested sodium hydroxide solution concentration.

The results trend is consistent with study and review done by Khale and Chaudhary (2007) which found that extended curing time enhance the polymerization process resulting in higher compressive strength.

4.2.5 Source of Raw Material

The source of raw materials is recognized as one of the contributing factor that slightly affects the compressive strength. From the study done by Galau and Ismail, the compressive strength of geopolymer cement containing POFA from different palm oil mill gives different range of results. It shows that POFA from different mill have different characteristic based on their mill operation. (Galau and Ismail, 2004)

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The compressive strength of POFA geopolymer cement is significantly influenced by the concentration of the NaOH solution. As the NaOH concentration increases, the compressive strength also increases. This might be due to the acceleration in the geopolymerization process with the increase of the NaOH concentration or molarity in certain curing time.

The curing time is also giving impact to the compressive strength. It is observed that, the longer the curing time, the higher the compressive strength. The longer curing time help in enhancing the polymerization process to occur.

By replacing OPC with POFA, it does help in reducing the by-product waste from palm oil factory. However, the additions of additives and other type of blended ashes may help in increasing the compressive strength of the geopolymer cement.

In conclusion, 100 percent POFA geopolymer cement might not be suitable to be used as cement replacement material. The objective to study the effects of the POFA as the raw materials for cementing and its cement properties is achieved.

5.2 Recommendations

The further research on POFA geopolymer cement should be continued because this technology will help in solves problems such as excessive agriculture by products and landfilling.

In the future, the study can be improved by adding other agricultural industrial wastes as new source of geopolymer cement. The addition of other Agro-Industrial Wastes may improve the properties of the geopolymer cement.

In addition, aside from testing the compressive strength of geopolymer cement, the thickening time and fluid loss should also be tested. The results can be compared with the physical requirement of API standard for oil cements to determine the suitability of the new geopolymer cement to be used as oil well cement.

REFERENCE

A.M Mustafa Al Bakri et al., (2011a). *Microstructure of different NaOH molarity of fly ash- based green polymeric cement*, 4.

A.M. MustafaAl Bakri et al., (2011b). *The Processing, Characterization and Properties of Fly Ash Based on Geopolymer Concrete*.

Abdullah, A. A. (1997). Palm Oil Shell Aggregate for Lightweight Concrete. In S. Chandra, *Waste Materials Used in Concrete Manufacturing* (p. 626). New Jersey: Noyes Publication.

Ali Hasanbeigi, L. P. (2012). CO2 Impact of Cement Production. *Emerging Energy-efficiency and CO_2 Emission-reduction Technologies for Cement and Concrete Production*, 5.

Amir H. Mahmoudkhani, S. D. (2008). SPE 115004.*New Environment-Friendly Cement Slurries With Enhanced Mechanical Properties*., 1-3.

Beijing Foresty and Parks Departments of Internation Coorperation. (2009, January 6). *Oil Palm Biomass*. Retrieved February 22, 2013, from BFPIC web site: http://www.bfdic.com/en/Features/Features/79.html

CCAA, (2006, April). *Cement Concrete Aggregated Australia.*. Retrieved February 15, 2013, from Curing of Concrete: http://www.concrete.net.au/publications/pdf/Curing06.pdf

Dam, T. J. (2010, March). *Geopolymer Concrete*. Retrieved February 16, 2013, from Tech Brief: http://www.fhwa.dot.gov/pavement/concrete/pubs/hif10014/hif10014.pdf

Davidovits, J. (1991). Geopolymers: Inorganic Polymeric New Materials. In J. Davidovits, *Geopolymer chemistry and applications* (pp. p.1633-1656). Saint Quentin, France: Institut Géopolymère.

Davidovits, J. (2013, February 6). Geopolymer Cement Review 2013. Retrieved February 16, 2013, from Geopolymer Institute: http://www.geopolymer.org/library/technical-papers/21-geopolymer-cement-review-2013

Galau, D. and Ismail, M. (2004). *Characterization of Palm Oil Fuel Ash (POFA) from Different Mill as Cement Replacement Material*, 2-7.

Faizal M.N. and Irawan S. (2007, July). Cementing Lecture Slides.

Fahmi Azrin, W.A. (2011). *Geopolymer Cement for Oil Well with Improved Properties. Department of Mechanical Engineering*, Final Year Project Dissertation, Universiti Teknologi PETRONAS, Malaysia.

Final Year Project Guidelines (2011). FYP Project Guidelines_v4

Hardjito,D. et al., (2004). ACI Materials Journal. *On the Development of Fly Ash-Based Geopolymer Concrete*, 3.

Hardjito,D. Cheak, C.C and Ho L.I.C., (2008). Modern Applied Science. *Strenght and Setting Times of Low Calcium Fly Ash-Based Geopolymer Mortar*, 4.

Instron. *Glossary for material testing*. (n.d.). Retrieved February 13, 2013, from Instron: http://www.instron.us/wa/glossary/Compressive-Strength.aspx

Institute, G. (2006, April 7). *Geopolymer Institute*. Retrieved April 13, 2013, from Geopolymer Institute Web Site: http://www.geopolymer.org/applications/global-warming

K.Abdullah. et al., (2006). *Pofa : A Potential Partial Cement Replacement.*

Khale, D. and Chaudhary, R. (2007). Mechanism of geopolymerization and factors influencing its development: a review.

Klee, H. (2009). The Cement Sustainability Initiative. *Cement Industry Energy and CO_2 Performance "Getting the Numbers Right,"*.

M. Azreen. Hussin, M.W and Bhutta, M.A.R (2011). Advanced Material Research. *Mix Design and Compressive Strength of Geopolymer Concrete Containing Blended Ash from Agro-Industrial Wastes*.

M. H. Ahmad et al., (2008). Compressive Strength of Palm Oil Fuel Ash Concrete.

McLeod, R. (2005). Ordinary Portland Cement With Extraordinary High CO2 Emission. What can be done to reduce them?

Mohamad H, H. H. (1986). Availability and Potential of Oil Palm Trunks and Fronds up to the year 2000. *Palm Oil Research Institute of Malaysia (PORIM)*, 1-6.

O. Abu Hassan, M. I. (1996). Oil Palm Fronds as a roughage feed source for ruminants in Malaysia. *Availability of Oil Palm Fronds*, 2-3.

P. Duxson, A. F.-J. (2006). Geopolymer technology: the current state of the art. 1-3.

Paloma A., Grutzeck.M.W. and Blanco M., (1998). Cement and Concrete Research. *Alkali-activated fly ashes, A cement for the future*.

Petermann, J. C., Saeed, A., & Hammond, M. I. (2010). Alkali-Activated Geopolymers: A Literature Review.

PetroWiki. (2012, September 12). *Cement composition and classification*. Retrieved July 3, 2013, from Petro Wiki: http://petrowiki.org/Cement_composition_and_classification#cite_ref-r1_0-3

Raw Polymer Limited. (2008). *Ordinary Portland Cemen Glossary*. Retrieved February 16, 2013, from http://www.ordinaryportlandcement.com/

Smith, D. K. (1987). *Cementing.* United States of America: Society of Petroleum Engineers.

Steven H. Kosmatka, B. K. (1988). Curing Concrete. In *Design and Control of Concrete Mixtures* (pp. 219-228).

System, G. C. (n.d.). *Gates Concrete.* Retrieved April 15, 2013, from Gates Concrete Web site: http://www.gatesconcreteforms.com/pdfs/slumpTest.pdf

Vanchai Sata, C. J. (2004). Utilization of Palm Oil Fuel Ash in High-Strength Concrete.

Yacob, S. (2007). *Progress and challenges in utilization of palm biomass.* Retrieved January 30, 2013, from http://www.jst.go.jp/asts/asts_j/files/ppt/15_ppt.pdf

APPENDIX

Appendix 1: Graphical experiment procedure

Weighing process

POFA

Sodium Hydroxide

Sodium Silicate

Mixing process

Materials used

Water is added

Cement slurry

Moulding process

7

Cement mould

8

Poured cement slurry

Curing process

9

Oven curing at 60°C

10

Ambient temperature curing

Testing process

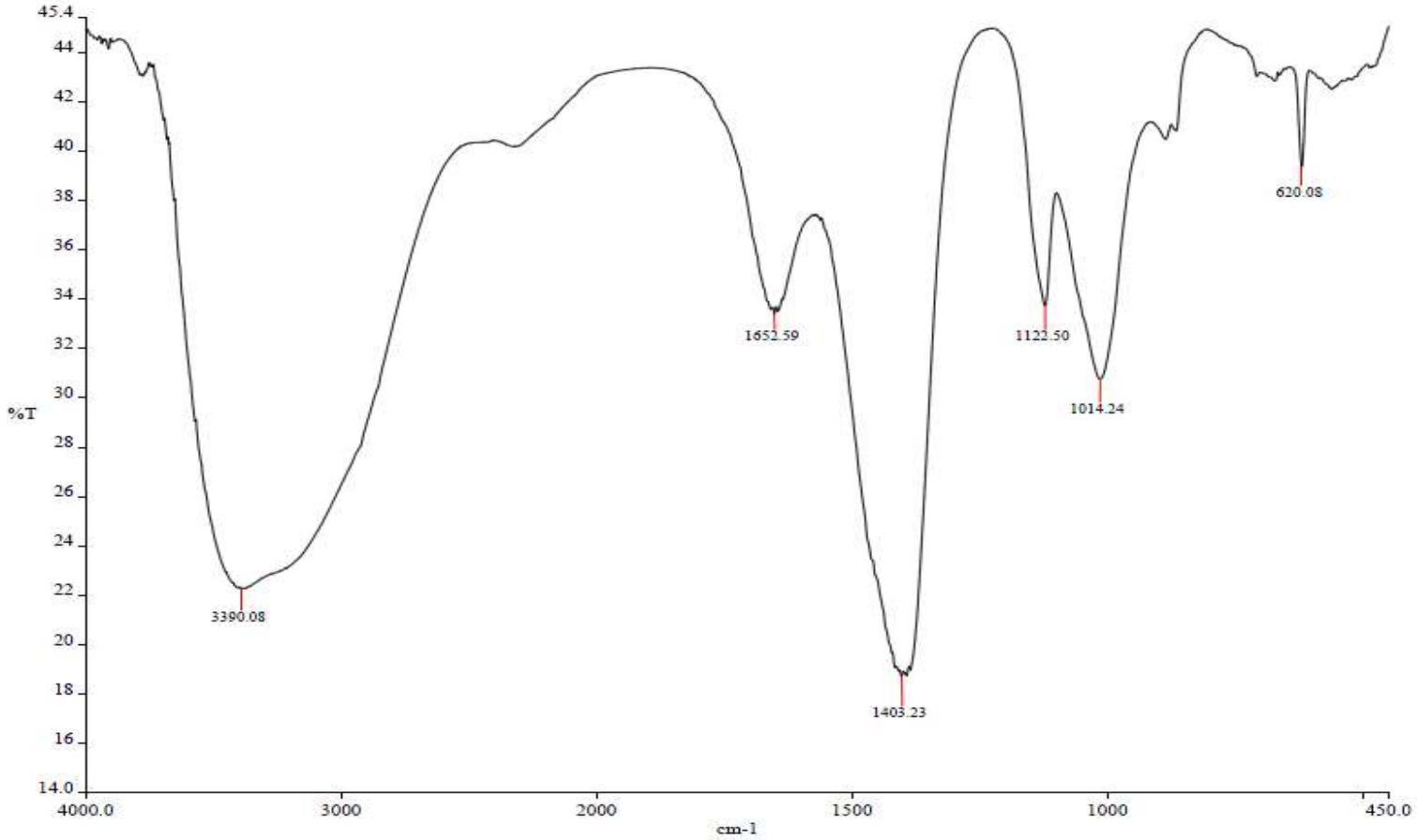
11

Before compressive test

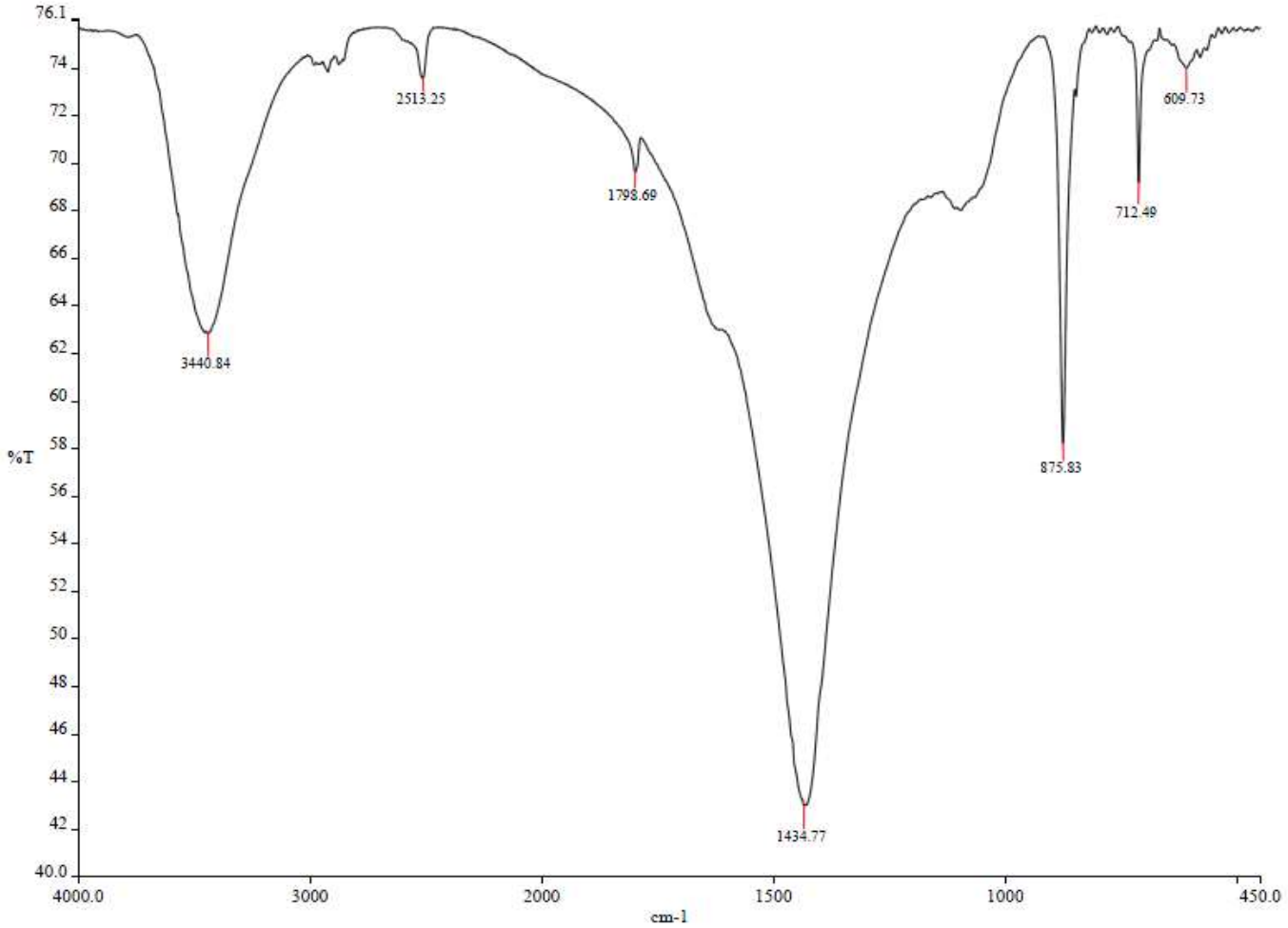
12

After compressive test

Appendix 2: FTIR test results (POFA and FA)



POFA FTIR result



FA FTIR result

Appendix 3

Infrared Spectroscopy

IR Absorptions for Representative Functional Groups

Functional Group	Molecular Motion	Wavenumber (cm^{-1})
alkanes	C-H stretch	2950-2800
	CH_2 bend	~1465
	CH_3 bend	~1375
	CH_2 bend (4 or more)	~720
alkenes	=CH stretch	3100-3010
	C=C stretch (isolated)	1690-1630
	C=C stretch (conjugated)	1640-1610
	C-H in-plane bend	1430-1290
	C-H bend (monosubstituted)	~990 & ~910
	C-H bend (disubstituted - E)	~970
	C-H bend (disubstituted - 1,1)	~890
	C-H bend (disubstituted - Z)	~700
	C-H bend (trisubstituted)	~815
alkynes	acetylenic C-H stretch	~3300
	C,C triple bond stretch	~2150
	acetylenic C-H bend	650-600
aromatics	C-H stretch	3020-3000
	C=C stretch	~1600 & ~1475
	C-H bend (mono)	770-730 & 715-685
	C-H bend (ortho)	770-735
	C-H bend (meta)	~880 & ~780 & ~690

	C-H bend (para)	850-800
alcohols	O-H stretch	~3650 or 3400-3300
	C-O stretch	1260-1000
ethers	C-O-C stretch (dialkyl)	1300-1000
	C-O-C stretch (diaryl)	~1250 & ~1120
aldehydes	C-H aldehyde stretch	~2850 & ~2750
	C=O stretch	~1725
ketones	C=O stretch	~1715
	C-C stretch	1300-1100
carboxylic acids	O-H stretch	3400-2400
	C=O stretch	1730-1700
	C-O stretch	1320-1210
	O-H bend	1440-1400
esters	C=O stretch	1750-1735
	C-C(O)-C stretch (acetates)	1260-1230
	C-C(O)-C stretch (all others)	1210-1160
acid chlorides	C=O stretch	1810-1775
	C-Cl stretch	730-550
anhydrides	C=O stretch	1830-1800&1775-1740
	C-O stretch	1300-900
amines	N-H stretch (1 per N-H bond)	3500-3300
	N-H bend	1640-1500
	C-N Stretch (alkyl)	1200-1025
	C-N Stretch (aryl)	1360-1250
	N-H bend (oop)	~800
amides	N-H stretch	3500-3180
	C=O stretch	1680-1630
	N-H bend	1640-1550

	N-H bend (1^{o})	1570-1515
alkyl halides	C-F stretch	1400-1000
	C-Cl stretch	785-540
	C-Br stretch	650-510
	C-I stretch	600-485
nitriles	C,N triple bond stretch	~2250
isocyanates	-N=C=O stretch	~2270
isothiocyanates	-N=C=S stretch	~2125
imines	R_2C=N-R stretch	1690-1640
nitro groups	-NO_2 (aliphatic)	1600-1530&1390-1300
	-NO_2 (aromatic)	1550-1490&1355-1315
mercaptans	S-H stretch	~2550
sulfoxides	S=O stretch	~1050
sulfones	S=O stretch	~1300 & ~1150
sulfonates	S=O stretch	~1350 & ~11750
	S-O stretch	1000-750
phosphines	P-H stretch	2320-2270
	PH bend	1090-810
phosphine oxides	P=O	1210-1140

(Source: http://www.chemistry.ccsu.edu/glagovich/teaching/316/ir/table.html)